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Systematic Analysis of the Effects of Moulding Conditions on the Properties of Shape Memory Polymers

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Abstract. Shape memory polymers (SMP) demonstrate a unique ability to recover to their original shape upon application of the external stimulus after being deformed and fixed into a temporary shape. The SMP part can be produced by injection moulding process but limited work has been done to understand the effects of moulding conditions on the shape memory effect. The aim of this research is to investigate the influence of selected moulding parameters on the shape memory effect (SME). Three moulding process parameters - injection speed, packing pressure and mould temperature were differentiated in order to produce the test parts. The samples were subjected to thermomechanical experiments and their shape before and after the experiments were analysed along with the overall quality of the parts. The results from these analyses are presented in the paper.

Keywords: Shape Memory Polymer, Shape Memory Effect, Injection Moulding Process, Thermo-mechanical Testing.

INTRODUCTION

Shape memory polymers (SMP) represent a very interesting class of so-called smart materials as they demonstrate a unique ability to recover to their original shape upon application of the external stimuli after being deformed and fixed into a temporary form. This mechanism is called shape memory effect (SME). In recent years SMP have received significant attention because of their flexibility, bio-compatibility, low cost, and ease of manufacturing together with their possibilities for many advanced applications. One of the main advantages is the ability to process SMP with conventional technology, such as injection moulding. In order to enhance their properties, their chemical composition has been constantly under development. Moreover several studies have been made in order to determine the influence on the deformation and recovery of the SMP by means of thermomechanical experiments. Various factors, such as environmental conditions, the deformation pattern, geometry of the samples, the molecular structures of the selected material etc. were studied [1, 2, 3, 4, and 5]. However, it is also necessary to characterise the influence of the processing methods and associated parameters on the recovery characteristics, which is an intermediate step between the material development and utilization. Moreover, the interplay between the overall quality of the parts and the shape memory properties need to be determined in order to achieve the best performance. This study focuses on establishment of the influence of the production process parameters on the shape memory characteristics and the resulting quality of the produced parts, by means of commonly used thermomechanical experiments and geometrical measurements of the shape of the specimens.

EXPERIMENTAL PROCEDURE

Specimens fabrication: Among commercial materials Thermoplastic Polyurethane (TPU) provided by BASF® was selected for the experiment. Glass transition of the material was 33 ºC. Two standard shapes of the specimens were chosen for the investigation: rectangular bar and dog-bone as shown in Figure 1.

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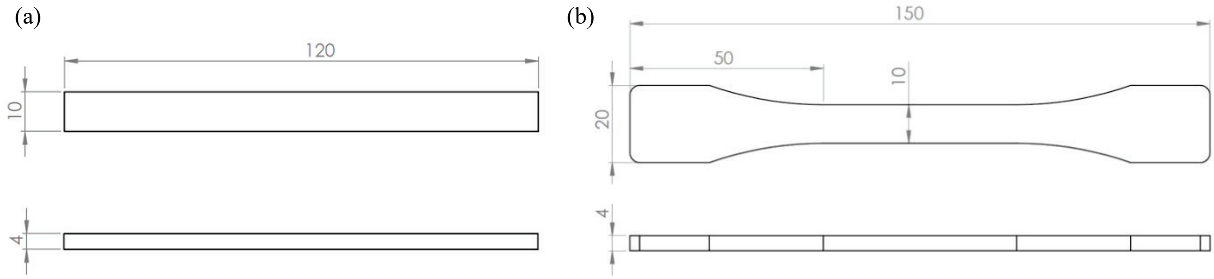

FIGURE 1. Drawings of the parts (dimensions in mm), rectangular-shaped specimen used for 3-point-bending test (a) and dog-bone-shaped specimen used for tensile test.

The parts were injection moulded using Milacron® Ferromatik K60 injection moulding machine. Process window was determined by means of simulation using Autodesk® Moldflow software. Table 1 shows the parameters selected and differentiated for the DOE analysis. Remaining moulding parameters were kept constant according to standard TPU process settings. The melt temperature was set to 235 ºC and the cycle time was 25 s with 20 s cooling time.

TABLE 1. Values of combined parameters.

Parameter	Low level	High level
Injection speed	30 mm/s	50 mm/s
Packing pressure	90 bar	110 bar
Mould temperature	20 ºC	40 ºC

Thermomechanical experiment

3-point-bending test for rectangular-shaped specimens was chosen in order to trigger the shape memory effect in TPU. The experiment was performed at MTS® 810 Material Testing System with climatic chamber and the maximum bending angle was set to 130º. Figure 2 depicts the course of the experiment. For a comparative study, the tensile test was performed for dog-bone-shaped samples at Instron® 8521 Material Testing System with climatic chamber. The maximum elongation of the sample was set to 55 mm. Both tests had the same patterns. First the specimens were heated to the deformation temperature, $T_d = T_g + 25$ ºC and deformed to the pre-determined strain. Next, they were cooled down under the load to fixing temperature, $T_f = T_g - 5$ ºC and held for 5 min. The specimens were then unloaded and heated to the recovery temperature, $T_r = T_g + 25$ ºC and also kept for 5 min. For each sample only one cycle was executed.

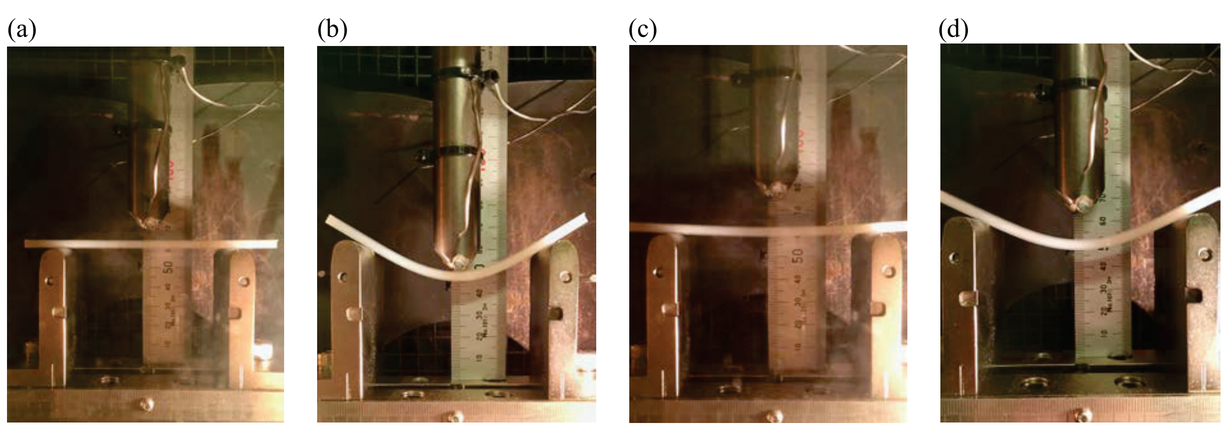

FIGURE 2. 3-point-bending experiment, stages: (a) the beginning of the experiment, (b) deformation in T_d and lowering the temperature to T_f, (c) unloading and (d) heating to T_r and recovery of the material.

Geometrical measurements

Both sets of specimens were measured before and after the thermomechanical experiments. For bending test, Computed Tomography (CT) was applied in order to achieve fast and automated measurements. CT scanning was performed at Nikon® XT H 225 ST system. The comparison was performed by measuring the angles of the samples before and after subjecting to bending, which is illustrated in Figure 3. The samples subjected to tension were measured by the calliper. The recovery ratio (R_R) was calculated according to formula (1) [5] and adjusted to the investigated values of bending angle for rectangular and length of the dog-bone-shaped samples accordingly.

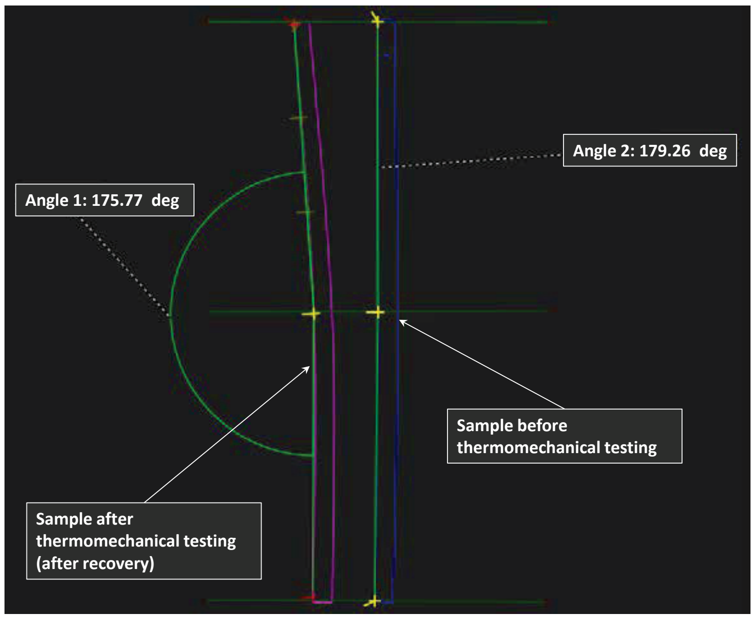

FIGURE 3. Angle measurement results from the rectangular bar before and after thermomechanical experiment.

The 3D scans were also used to assess the overall quality of the parts. This was performed by comparing the obtained geometry of the rectangular samples to the CAD design of the parts. The quality of the parts was evaluated by the percentage of the geometry of the part which falls into the pre-determined deviation threshold of ± 200 μm.

$$R_R = \frac{\varepsilon_m - \varepsilon_p(N)}{\varepsilon_m - \varepsilon_p(N-1)} \quad (1)$$

RESULTS

Recovery ratio (R_R): Due to the differences in the measuring methods, samples geometry, and the applied deformation, the absolute values cannot be compared. However the obtained R_R revealed that significant increase of the recovery ratio in case of both bending and tensile test occurs when the samples are produced with higher injection speed. This is evident when looking at the main effects plots, which are shown in Figure 4.

(a) (b)

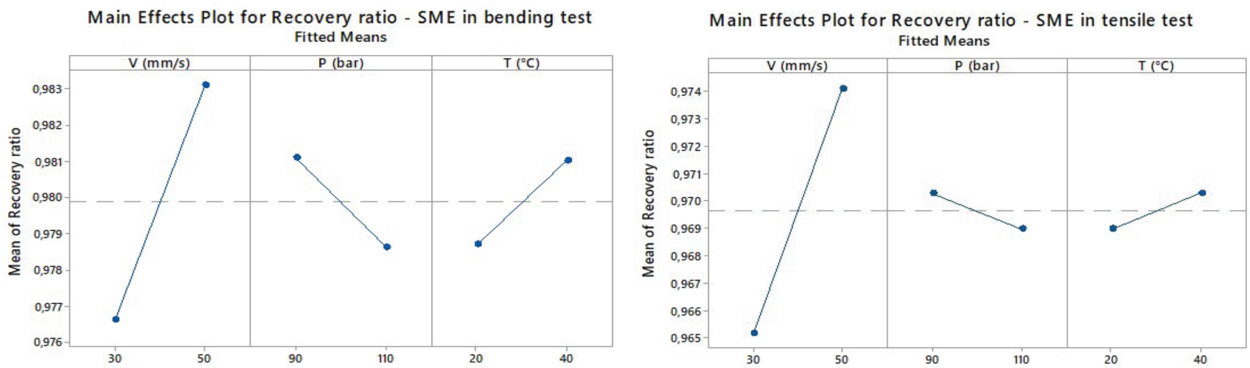

FIGURE 4. Main effects plots of the recovery ratio of the samples after bending and tensile experiments for injection velocity, packing pressure and mould temperature (abbreviated as V, P and T respectively).

This phenomenon was associated to the effect of molecular orientation, which increases with the raised shear rate due to the higher injection speed. This was confirmed by the results of the values of the maximum shear rate obtained by means of simulation with fixed remaining parameters, as illustrated in Table 2. High orientation increases the hard segments aggregation, which enhances the recovery force at elevated temperature [6]. Moreover slight increase of recovery was observed with decreasing packing pressure and increasing mould temperature. This can be explained by occurrence of residual stresses which are a driving force for shape memory effect [7]. Residual stresses are higher at low packing pressures and high mould temperatures which is in agreement with obtained $R_{R.}$ The dependency of the recovery ratio on the packing pressure and mould temperature should be however further investigated, as the differences in the results are not significant. Nevertheless, both 3-point-bending and tensile tests resulted in recovery ratio higher that 95 %, which revealed the excellent shape memory characteristics of the chosen TPU.

TABLE 2. Dependency of the shear rate on the injection speed.

Shape	Injection velocity	Shear rate
Rectangular bar	30 mm/s	170 1/s
	50 mm/s	208.8 1/s
Dog-bone	30 mm/s	232.7 1/s
	50 mm/s	285.1 1/s

Dimensional accuracy of the parts: As it can be seen in Figure 5, it is evident that the dimensions fall in the set interval in the highest amount when the packing pressure is increased. This is contradictory to the results of the shape memory effect, where the packing pressure had the detrimental effect on the performance. This phenomenon can be also explained as the effect of the residual stresses, which, when they are high, induce the propagation of warpage and shrinkage of the part [8]. The increased conformance of geometry with increased mould temperature of the part however cannot be explained by the occurrence of residual stresses. The mould temperature has a prominent impact on the overall shrinkage of the parts which hinders the detrimental effect, caused by the increase of residual stresses.

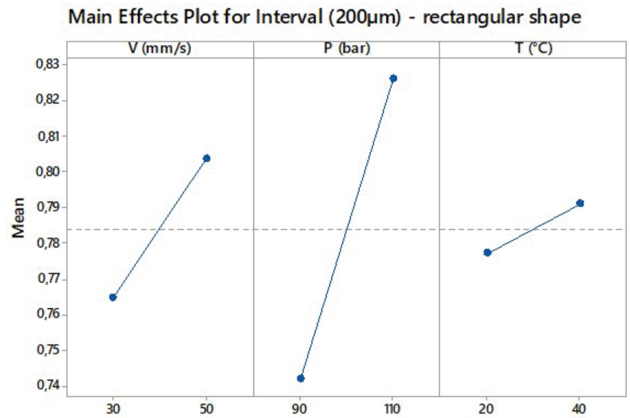

FIGURE 5. Main effects plot of the conformation of the rectangular-shaped parts with the respect to the CAD design for injection velocity, packing pressure and mould temperature, abbreviated as V, P and T respectively.

CONCLUSIONS

The outcome of the research showed that injection speed and mould temperature has an advantageous influence on the thermally-induced recovery characteristics and overall quality of the parts produced with injection moulding. The third investigated parameter, i.e. packing pressure has however contradictory effects on the recovery ratio and overall quality of the parts. The results show that the shape memory effect is mostly governed by the molecular orientation of the polymer along with residual stresses. From the manufacturing point of view, it is therefore of great importance to collectively analyse the influential parameters when designing parts which should exhibit the shape memory characteristics together with the dimensional accuracy. As future continuation of this work, it is suggested to investigate the other relevant moulding parameters and different SMP in order to establish a wide range of the process window for the highest recovery ratio in general. An increased interval of the selected process parameters for DOE analysis could be advantageous to achieve a higher recovery effect. Moreover the investigation of molecular orientation and fiber orientation in case of fiber filled materials are advised in order to confirm their influence on the shape memory effect.

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